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(54) Title: SPILL RESISTANT CARPET BACKING			
(57) Abstract			
<p>An aqueous dispersed polymeric composition for preparing the spill resistant carpet backing is provided. The aqueous dispersed polymeric composition of the present invention comprises an aqueous dispersed polymeric material, an inorganic filler, and a hydrophobic compound selected from the group consisting of a hydrophobic acid, a salt of a hydrophobic acid, and mixtures thereof. Alternatively, the inorganic filler is pretreated with the hydrophobic compound. Also, a kit and a method for preparing a spill resistant carpet backing from the aqueous dispersed polymeric composition is provided.</p>			

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**SPILL RESISTANT CARPET BACKING****FIELD OF THE INVENTION**

This invention relates to a spill resistant carpet backing. More particularly, it relates to using a hydrophobic acid-treated inorganic filler or salts thereof as part of an aqueous dispersed polymeric composition to increase the spill resistance of a carpet backing.

**DESCRIPTION OF THE PRIOR ART**

Generally, tufted carpets minimally consist of tufted fibers through a primary backing and a precoat. Tufted carpets have a precoat and may also have additional layers such as a laminate layer, a secondary layer, and a foam layer. Moreover, the tufted carpet may have more than one secondary layer. The tufted carpet may be applied to a variety of subfloors, including wood, concrete, and tile.

The precoat is required to anchor the carpet tufts to the primary backing. It may also contain an adhesive to adhere the tufted carpet to additional layers or the subfloor. The precoat can affect the carpet's tuft bind, hand, delaminating properties, wet strength properties, wear resistance, and barrier performance. Alternatively, a laminate layer may be applied without a precoat and anchor the carpet tufts to the primary backing. But, better

anchoring is achieved when a precoat is applied than with a laminate layer is applied alone.

The precoat may be prepared from several materials. However, it is usually prepared from a polyurethane material or a styrene-butadiene latex. It may be prepared from a butadiene-acrylonitrile latex, an ethylene-vinyl acetate latex, a styrene-butadiene-butyl acrylate latex, a chloroprene latex, a polyethylene copolymer latex, an ethylene-styrene latex, a styrene-butadiene-vinylidene chloride latex, a styrene-alkyl acrylate latex, a vinyl latex, or an acrylic latex.

With regard to a polyurethane precoat, conventional practice in the carpet manufacturing industry requires that the precoat be prepared from an isocyanate formulation (A-side formulation) and a polyol formulation (B-side formulation) at the carpet manufacturing site. This is sometimes referred to as "A+B chemistry". Preparing a polyurethane precoat by A+B chemistry can result in unpredictable loss of production and inefficiency due to problems that can occur in carrying out the reaction at the manufacturing site, such as premature gellation.

Alternatively, the polyurethane precoat may be applied as an aqueous polyurethane (PU) dispersion. Aqueous PU dispersions can be prepared by polymerizing the polyurethane reactants in an organic solvent followed by dispersion of the resulting solution in water, and optionally followed by removal of organic solvent. See U.S. Patent Nos. 3,437,624; 4,092,286; 4,237,264; 4,742,095; 4,857,565; 4,879,322; 5,037,864; and 5,221,710, which are incorporated herein by reference. Also, an aqueous polyurethane dispersion may be prepared by first forming a prepolymer, next dispersing the prepolymer in water, and finally conducting a chain extension in the water as disclosed in WO 98/41552, published September

24, 1998, which is incorporated herein by reference. Preparations of aqueous dispersions of polyurethane are also described in U. S. Patent Application Serial Nos. 09/039,978 and 09/039,976. U.S. Patent No. 4,296,159 to Jenkins, et al., discloses preparing a tufted or woven article having a unitary backing prepared by applying a polyurethane forming composition to the underside of the tufted or woven article.

The precoat may consist of an aqueous styrene butadiene latex. Styrene-butadiene (SB) latexes for use in carpet are described, for example, in P.L. Fitzgerald, "Integral Latex Foam Carpet Cushioning", J. Coat. Fab. 1977, Vol. 7 (pp.107 - 120); and in R.P. Brentin, "Latex Coating Systems for Carpet Backing", J. Coat. Fab. 1982, Vol. 12 (pp. 82 - 91). SB latexes provide flexibility in production costs owing to the ability to include low to high concentrations of filler component in a low viscosity latex. However, SB latexes with filler may not meet the rigorous standards set for intermediate grade carpets. In addition, current technology may require that a latex material remain stable for a period of up to one year. High solids content affects the stability of SB latexes. Accordingly, commercially available SB latexes typically do not have a solids content of greater than 55%.

Without regard to the type of material used to make the precoat, a liquid spill can detrimentally affect the carpet's performance and appearance. The liquid can flow through the precoat and then onto, or be absorbed by, the underside of the carpet. In particular, spilled liquids such as beverage drinks, food, blood, urine, and feces can penetrate to the underside of the carpet or into the subfloor so that the liquids can be inaccessible by various cleaning methods.

When the precoat is an aqueous polymeric dispersion, various conventional methods

are used to provide a moisture or spill resistant barrier between the primary backing and other layers or the subfloor. The conventional methods include (1) modifying the precoat formulation, (2) adding an essentially impermeable membrane between the precoat and the other layers or the subfloor, and (3) applying a spill resistant coating onto the precoat or 5 another layer.

An example of an attempt to improve spill resistance by modifying the precoat formulation is increasing the coating weight of the dispersion. This method is undesirable because it requires more material, which is expensive. It can also yield a final carpet having undesirable hand or stiffness properties. Moreover, the precoat has an increased probability 10 of blistering in the drying ovens.

Another example of an attempt to improve spill resistance by modifying the precoat formulation is adding a wax filler to the dispersion. While the addition of waxes to aqueous styrene-butadiene latexes is known to improve the water barrier properties of the precoat, it is also known that wax additives are generally detrimental to other carpet properties.

15 Another example of a formulation modification is the reduction of filler levels. But, this formulation change increases the carpet cost and may increase the tendency of the precoat to blister in the drying ovens.

Examples of an attempt to improve spill resistance by adding an essentially impermeable membrane between the precoat and the other layers or the subfloor are 20 described in U. S. Patent Nos. 4,336,089 and 5,763,040. In U.S. Patent No. 4,336,089, the patentees describe a water-impervious film that collects the spilled liquid below the carpet surface. In U.S. Patent No. 5,763,040, the patentees describe applying a non-permeable

fabric or film to a carpet backing. This secondary backing prevents liquid flow to subsequent layer or to the subfloor. Unfortunately, both described membranes may permit the spilled liquid to collect at a depth between the carpet and the underlay barrier such that the spilled liquid cannot be removed through traditional cleaning methods.

5 An example of an attempt to improve spill resistance by applying a spill resistant coating is applying a fluorochemical or another material onto the precoat or another layer. U.S. Patent No. 5,348,785 describes applying a fluorochemical on the underside of a secondary backing. The use of fluorochemicals to impart water impermeability is also described in U.S. Patent Nos. 4,619,853 and 4,643,930. Unfortunately, the use of  
10 fluorocarbons can increase the cost of the carpet. Additionally, steam or extraction cleaning of a carpet can leach the fluorocarbons out of the carpet.

Other materials that are applied as a spill resistant coating include silicone-based compositions, wax emulsions, naturally occurring oils, and hydrophobic acrylate resins.  
15 Applying these various hydrophobic compositions to a secondary backing is described in U. S. Patent No. 5,558,916, but these compositions do not prevent absorption of spilled liquids by underlying layers.

It would be desirable to prepare a carpet backing that resists penetration by spilled liquids. It would also be desirable to prepare the carpet backing by a process that would not significantly increase the cost of manufacturing the carpet. Further, it would be desirable to  
20 prepare a spill resistant carpet backing that can protect the underside of a carpet from spills such that the spilled liquids would be accessible for removal by various cleaning methods.

## SUMMARY OF THE INVENTION

According to the present invention, a spill resistant carpet backing is provided. More specifically, an aqueous dispersed polymeric composition for preparing the spill resistant carpet backing is provided.

5 In the preferred embodiment, the aqueous dispersed polymeric composition of the present invention comprises an aqueous dispersed polymeric material, an inorganic filler, and a hydrophobic compound selected from the group consisting of a hydrophobic acid, a salt of a hydrophobic acid, and mixtures thereof. Alternatively, the inorganic filler is pretreated with the hydrophobic compound. Also, a kit and a method for preparing a spill resistant carpet  
10 backing from the aqueous dispersed polymeric composition is provided. While the most significant impact on spill resistance is achieved through using the aqueous dispersed polymeric composition as a precoat, the aqueous dispersed composition may be applied as a laminate layer or a foam layer to improve spill resistance.

## DESCRIPTION OF THE INVENTION

15 In the preferred embodiment of the present invention, the aqueous dispersed polymeric composition comprises an aqueous dispersed polymeric material, an inorganic filler, and a hydrophobic compound selected from the group consisting of a hydrophobic acid, a salt of a hydrophobic acid, and mixtures thereof. The aqueous dispersed polymeric material is selected from the group consisting of a polyurethane dispersion, a styrene-  
20 butadiene latex, a butadiene-acrylonitrile latex, an ethylene-vinyl acetate latex, a styrene-butadiene-butyl acrylate latex, a chloroprene latex, a polyethylene copolymer latex, an ethylene-styrene latex, a styrene-butadiene-vinylidene chloride latex, a styrene-alkyl acrylate

latex, a vinyl latex, an acrylic latex, and mixtures thereof. The preferred aqueous dispersed polymeric composition is a polyurethane dispersion.

In the present invention, polyurethane can refer to a polyurethane compound, a polyurea compound, or mixtures thereof. A polyurethane compound can be obtained by the 5 reaction of a polyol with a polyisocyanate. A polyurea compound can be obtained by the reaction of an amine with a polyisocyanate. A polyurethane compound or polyurea compound can contain both urea and urethane functionality, depending on what compounds are included in the A and/or B-side formulations. For the purposes of the present application, no further distinction will be made herein between the polyurethane compounds and polyurea 10 compounds. The term "polyurethane" will be used generically to describe a polyurethane compound, a polyurea compound, and mixtures thereof.

A polyurethane dispersion composition useful in the practice of the present invention includes water, and a polymeric compound selected from the group consisting of a polyurethane compound, a mixture of polyurethane-forming compounds, and mixtures 15 thereof. A polyurethane dispersion as described herein can include chain extenders, surfactants, fillers, dispersants, foam stabilizers, thickeners, fire retardants, defoamers, and other materials useful in polyurethane formulations.

Polyurethane-forming compounds as used in the present invention are compounds that are capable of forming polyurethane polymers. Polyurethane-forming compounds include, 20 for example, polyurethane prepolymers. Prepolymers useful in the practice of the present invention are prepared by the reaction of active hydrogen compounds with any amount of isocyanate in excess material relative to active hydrogen material. The isocyanate

functionality can be present in an amount of from about 0.2 wt% to about 40 wt%. A suitable prepolymer can have a molecular weight in the range of from about 100 to about 10,000. Prepolymers useful in the practice of the present invention should be substantially liquid under the conditions of dispersion.

5 Active hydrogen compounds can be described as compounds having functional groups that contain at least one hydrogen atom bonded directly to an electronegative atom such as nitrogen, oxygen or sulfur. Suitable active hydrogen compounds can be polyols of molecular weight of less than about 6000.

When the polymeric material is a polyurethane compound, the aqueous dispersed  
10 polymeric composition can include a chain extender. A chain extender is used herein to build the molecular weight of the polyurethane prepolymer by reaction of the chain extender with the isocyanate functionality in the polyurethane prepolymer, i.e., chain extend the polyurethane prepolymer. A suitable chain extender is typically a low equivalent weight active hydrogen containing compound, having about 2 or more active hydrogen groups per  
15 molecule. The active hydrogen groups can be hydroxyl, mercaptyl, or amino groups. An amine chain extender can be blocked, encapsulated, or otherwise rendered less reactive. Other materials, particularly water, can function to extend chain length and so are chain extenders for purposes of the present invention. Polyamines are preferred chain extenders. It is particularly preferred that the chain extender be selected from the group consisting of  
20 amine terminated polyethers such as, for example, Jeffamine D-400 from Huntsman Chemical Company, amino ethyl piperazine, 2-methyl piperazine, 1,5-diamino-3-methylpentane, isophorone diamine, ethylene diamine, diethylene triamine, triethylene tetramine,

triethylene pentamine, ethanol amine, lysine in any of its stereoisomeric forms and salts thereof, hexane diamine, hydrazine and piperazine. In the practice of the present invention, the chain extender is often used as a solution of chain extender in water.

Small amounts of chain extender can be advantageously used. Generally, the chain extender is employed at a level sufficient to react with from about zero (0) to about 100 percent of the isocyanate functionality present in the prepolymer, based on one equivalent of isocyanate reacting with one equivalent of chain extender. It can be desirable, under certain conditions, to allow water to act as a chain extender and react with some or all of the isocyanate functionality present.

A catalyst can be used to promote the reaction between a chain extender and an isocyanate. Suitable catalysts include tertiary amines, organometallic compounds, similar compounds, and mixtures thereof. For example, suitable catalysts include di-n-butyl tin bis(mercaptopropionic acid isoctyl ester), dimethyltin dilaurate, dibutyltin dilaurate, dibutyltin sulfide, stannous octoate, lead octoate, ferric acetylacetone, bismuth carboxylates, triethylenediamine, N-methyl morpholine, similar compounds, and mixtures thereof. An amount of catalyst is advantageously employed such that a relatively rapid cure to a tack-free state can be obtained. If an organometallic catalyst is employed, such a cure can be obtained by using from about 0.01 to about 0.5 parts per 100 parts of the polyurethane-forming composition, by weight. If a tertiary amine catalyst is employed, the catalyst preferably provides a suitable cure using from about 0.01 to about 3 parts of tertiary amine catalyst per 100 parts of the polyurethane-forming composition, by weight. Both an amine type catalyst and an organometallic catalyst can be employed in combination.

The present invention can include other filler materials. The filler material can include conventional fillers such as milled glass, calcium carbonate, aluminum trihydrate, talc, bentonite, antimony trioxide, kaolin, fly ash, or other known fillers. A suitable filler loading in a polyurethane dispersion can be from about 100 to about 1000 parts of filler per 5 100 parts of the polyurethane compound. Preferably, the filler material can be loaded in an amount of at least about 200 pph (phr), more preferably at least about 300 pph, most preferably at least about 400 pph.

The present invention can include a filler wetting agent. A filler wetting agent generally renders the filler material compatible with a polyurethane-forming composition.

10 Useful wetting agents include phosphate salts such as sodium hexametaphosphate. A filler wetting agent can be included in a polyurethane-forming composition of the present invention at a concentration of at least about 0.5 parts per 100 parts of filler, by weight.

The present invention can include other components, such as surfactants, blowing agents, frothing agents, defoamers, fire retardants, pigments, antistatic agents, reinforcing 15 fibers, antioxidants, preservatives, acid scavengers, and the like. Useful surfactants include cationic and anionic surfactants. Examples of anionic surfactants include sulfonates, carboxylates, and phosphates. Examples of cationic surfactants include quaternary amines.

Surfactants can be either external or internal. External surfactants are surfactants are not chemically reacted into the polymer during dispersion preparation. Internal surfactants 20 are chemically reacted into the polymer during dispersion preparation. A surfactant can be included in an amount ranging from about 0.01 to about 20 parts per 100 parts by weight of polyurethane component.

Examples of suitable blowing agents are gases such as air, carbon dioxide, nitrogen, argon, and helium; liquids such as water and volatile halogenated alkanes; and azo-blowing agents such as azobis(formamide). Volatile halogenated alkanes include the various chlorofluoromethanes and chlorofluoroethanes. The use of a gas as a blowing or frothing agent is preferred. Particularly preferable is the use of air as a blowing or frothing agent. A frothing agent can differ from a blowing agent in that frothing agents are typically introduced by mechanical introduction of a gas into a liquid to froth the polymeric composition.

A polyurethane dispersion of the present invention can be stored for later application to the back of a carpet. Storage for this purpose requires that the dispersion be storage-stable.

Alternatively, the polyurethane dispersion can be applied in a continuous manner to the back of a carpet primary backing. That is, the dispersion can be applied to the back of a carpet as the dispersion is obtained. Polyurethane dispersions applied to a carpet in a continuous manner are not required to be storage-stable, and can have higher solids content and/or larger mean particle size than typical storage-stable polyurethane dispersion formulations.

A suitable storage-stable polyurethane dispersion as defined herein is any polyurethane dispersion having a mean particle size of less than about 5 microns; a more preferable mean particle size is less than about 1 micron. A polyurethane dispersion that is not storage-stable can have a mean particle size of greater than 5 microns. For example, a suitable dispersion can be prepared by mixing a polyurethane prepolymer with water and dispersing the prepolymer in the water using a commercial blender. Alternatively, a suitable dispersion can be prepared by feeding a prepolymer into a static mixing device along with water, and dispersing the water and prepolymer in the static mixer. Continuous methods for

preparing aqueous dispersions of polyurethane are known and can be used in the practice of the present invention. For example, U.S. Patent Nos. 3,437,624; 4,092,286; 4,237,264; 4,742,095; 4,857,565; 4,879,322; 5,037,864; and 5,221,710 describe continuous processes useful for obtaining aqueous polyurethane dispersions. In addition, a continuous process for 5 preparing an aqueous polyurethane dispersion having a high internal phase ratio is described in U.S. Patent No. 5,539,021, incorporated herein by reference.

The steps used in preparing a polyurethane carpet backing can be carried out in a continuous manner. For example, the prepolymer can be prepared from a suitable active hydrogen-containing compound in a continuous manner. The prepolymer can be loaded 10 directly into a mixing device with water to obtain an aqueous dispersion. Ultimately, the aqueous polyurethane dispersion can be applied to a carpet primary backing in a continuous manner to obtain a polyurethane-backed carpet.

In preparing polyurethane-backed carpets according to the present invention, an aqueous polyurethane dispersion is applied as a layer of preferably uniform thickness onto 15 one surface of a carpet primary backing. Aqueous polyurethane dispersions of the present invention can be applied as a precoat, a laminate layer, or a foam layer. Polyurethane precoats, laminate layers, and foam layers can be prepared by methods known in the art. Precoats, laminate layers and foam layers prepared from latexes are described in P. L. Fitzgerald, "Integral Latex Foam Carpet Cushioning", J. Coat. Fab. 1977, Vol. 7 (pp.107 - 20 120), and in R. P. Brentin, "Latex Coating Systems for Carpet Backing", J. Coat. Fab. 1982, Vol. 12 (pp. 82 - 91). In preparing a frothed polyurethane backing (frothing), it is preferred to mix all components and then blend a gas into the mixture, using equipment such as an

Oakes or Firestone foamer.

The polyurethane-forming composition can be applied to one surface of a carpet primary backing before it cures to a tack-free state. Alternatively, a polyurethane dispersion containing no unreacted isocyanate functionality can be applied, thereby removing the need 5 to cure the polymer. Typically the polyurethane-forming composition is applied to the surface attached to a primary backing.

The composition may be applied and gauged to the carpet primary backing using equipment such as a doctor knife, air knife, or extruder. Alternatively, the composition may be applied by (1) forming it into a layer on a moving belt or other suitable apparatus, (2) 10 dehydrating or partially curing, and (3) finally, marrying it to the carpet primary backing. It may be married to the carpet primary backing with equipment such as a double belt laminator (also known as double band) or a moving belt with an applied foam cushion.

The amount of polyurethane-forming composition used can vary widely, from about 5 to about 500 ounces per square yard, depending on the characteristics of the textile. After the 15 layer is applied and gauged, water is removed from the dispersion and the layer can be cured using heat from any suitable heat source such as an infrared oven, a convection oven, or heating plates.

A styrene-butadiene latex useful for practicing the present invention is disclosed in P.L. Fitzgerald, "Integral Latex Foam Carpet Cushioning", J. Coat. Fab. 1977, Vol. 7 (pp.107 20 - 120) and in R.P. Brentin, "Latex Coating Systems for Carpet Backing", J. Coat. Fab. 1982, Vol. 12 (pp. 82 - 91), both incorporated herein by reference. A styrene-butadiene-vinylidene

chloride latex useful for practicing the present invention is disclosed in U.S Patent No. 5,741,393, incorporated herein by reference.

U.S. Patent No. 5,770,660, incorporated herein by reference, discloses a method for preparing a butadiene-acrylonitrile latex that is useful for practicing the present invention.

5 U.S. Patent Nos. 3,644,262 and 4,164,489, incorporated herein by reference, disclose methods for preparing ethylene-vinyl-acetate latexes that are useful for practicing the present invention. U.S. Patent No. 5,591,806, incorporated herein by reference, discloses a method for preparing an ethylene acrylic acid aqueous dispersion that is useful for practicing the present invention.

10 U.S. Patent Nos. 3,890,261 and 5,661,205, incorporated herein by reference, disclose methods for preparing chloroprene latexes useful for practicing the present invention. U.S. Patent No. 5,380,785, incorporated herein by reference, discloses a method for preparing a butyl acrylate-acrylonitrile-styrene latex that is useful for practicing the present invention.

15 U.S. Patent Nos. 4,689,256 and 5,300,551, incorporated herein by reference, disclose methods for preparing vinyl chloride polymer latexes that are useful in practicing the present invention.

U.S. Patent No. 5,591,806, incorporated herein by reference, discloses a method for preparing a ethylene acrylic acid copolymer latex that is useful in practicing the present invention. U.S. Patent No. 4,714,728, incorporated herein by reference, discloses a method 20 for preparing an aqueous dispersion of acidic ethylene interpolymers that are useful in practicing the present invention.

U.S. Patent No. 4,228,058, incorporated herein by reference, discloses a method for preparing a mixture of vinylidene chloride latex and a styrene-butadiene latex useful in practicing the present invention. Other methods of preparing useful aqueous dispersed polymeric compositions would be readily apparent to a person of ordinary skill in the art in view of this disclosure. Those methods and aqueous dispersed polymeric compositions are within the scope of the invention with regard to preparing useful aqueous dispersed polymeric compositions.

The inorganic fillers of the present invention include calcium carbonate, calcium sulfate, kaolin, lignite fly ash, silica, talc, feldspar, mica, glass spheres, wollastonite, aluminum trihydrate, aluminum oxide, fiber glass, similar compounds, and mixtures thereof. In the present invention, the preferred inorganic filler is calcium carbonate.

The hydrophobic acids and salts of hydrophobic acid for treating the inorganic filler mixtures of the present invention include C4 to C22 acids and salts thereof. Also, the acids can be saturated or unsaturated. Examples of such acids include, but are not limited to, butyric acid, hexanoic acid, octanoic acid, decanoic acid, dodecanoic acid, lauric acid, myristic acid, palmitic acid, oleic acid, linoleic acid, stearic acid, linolenic acid, similar compounds, and mixtures thereof. In the present invention, the preferred acid-treated inorganic filler is stearic acid-treated calcium carbonate.

The salts of any of the hydrophobic acids described herein, alone or in admixture with any other salt or hydrophobic acid, can be useful in the practice of the present invention. Preferably, the salt is selected from the group consisting of calcium stearate, lithium stearate, and zinc stearate, and more preferably, zinc stearate, and most preferable, wettable zinc

stearate. Wettable zinc stearate is easily and uniformly compounded with a polyurethane dispersion and yields superior spill resistance. The preferred inorganic filler and hydrophobic salt combination is calcium carbonate with zinc stearate.

Other useful hydrophobic acids or hydrophobic acid salts include gum rosin, wood 5 rosin, tall oil rosin, abietic acid, oxidized polyethylene containing carboxylic acid groups, ethylene-acrylic acid copolymers, ethylene-methacrylic acid copolymers, polyolefins grafted with unsaturated carboxylic acids, polyolefins grafted with anhydrides such as maleic anhydride, methacrylic acid, maleic acid, fumaric acid, acrylic acid, and the like.

The hydrophobic acid or a salt thereof can be added to the carpet backing formulation 10 mixture in an amount of from about 0.025% weight by weight (w/w) to about 10% (w/w), based on the total weight of solids present in the mixture. Preferably the hydrophobic acid or its salt is added in an amount of from about 0.5% (w/w) to about 6.0% (w/w), more preferably from about 1.0% (w/w) to about 3.0% (w/w) most preferably from about 1.0% (w/w) to about 2.0% (w/w), based on total solids.

15 The inorganic filler can also be surface treated with the hydrophobic acid or its salt prior to introduction into the carpet backing dispersion formulation. This may be accomplished in a variety of ways. For example, the hydrophobic acid may be surface coated onto the filler in a high intensity mixer above the melting point of the hydrophobic acid. Another method involves the spraying of the aqueous dissolved or dispersed hydrophobic 20 acid or its salt onto the agitated filler.

In an alternate embodiment, the aqueous dispersed polymeric composition comprises an aqueous dispersed polymeric material and a treated inorganic filler, having been treated

with a hydrophobic compound selected from the group consisting of a hydrophobic acid, a salt of a hydrophobic acid, and mixtures thereof.

In another alternate embodiment of the present invention, a kit for preparing a spill resistant carpet backing is provided. It comprises an aqueous dispersed polymeric material, 5 an inorganic filler, and a hydrophobic compound selected from the group consisting of a hydrophobic acid, a salt of a hydrophobic acid, and mixtures thereof. Alternatively, the kit can comprise an aqueous dispersed polymeric material and a treated inorganic filler, having been treated with a hydrophobic compound selected from the group consisting of a hydrophobic acid, a salt of a hydrophobic acid, and mixtures thereof.

10 The present invention also provides a method for preparing a spill resistant carpet backing comprising (a) mixing an inorganic filler with an aqueous dispersed polymeric material and (b) admixing a hydrophobic compound selected from the group consisting of a hydrophobic acid, a salt of a hydrophobic acid, and mixtures thereof. Alternatively, the method can comprise (a) treating an inorganic filler with a hydrophobic compound selected 15 from the group consisting of a hydrophobic acid, a salt of a hydrophobic acid, and mixtures thereof and (b) mixing the treated inorganic filler with an aqueous dispersed polymeric material.

## EXAMPLES

The following examples merely exemplify various embodiments of the invention. It 20 is understood that the following examples are provided to further illustrate the invention. They do not in anyway limit the scope of the present invention.

Table of Compounds

The following compounds were used in the preparation of the exemplified dispersions. The table provides the compound's generic name, its trade mark name, and its vendor.

Generic Name	Trade Mark	Vendor
ethylene oxide capped polypropylene oxide diol, 2000 molecular weight	VORANOL™ 5287 Polyol	The Dow Chemical Company
50:50 mixture of 2,4'-diisocyanatodiphenylmethane and 4,4'-diisocyanatodiphenylmethane	ISONATE™ 50 MDI	The Dow Chemical Company
25:75 mixture of 2,4'-diisocyanatodiphenylmethane and 4,4'-diisocyanatodiphenylmethane	ISONATE™ 25 OP MDI	The Dow Chemical Company
Dispersing surfactant	DeSulf DBS-60T	Deforest Enterprises Inc.
Hydrocarbon-based defoamer	Nopco NDW	Henkel Corp.
Silicon-based aqueous dispersions	DC 85	Dow Corning Corp
Calcium carbonate	Georgia Marble #8	Georgia Marble Co.
Stearic acid treated calcium carbonate	Georgia Marble CS11	Georgia Marble Co.
Aqueous ethylene acrylic acid dispersion	Michem P4983-40R	Michem
Liquid ammonium stearate (33%)	STANFAX 320	Parachem Southern Inc.
Zinc stearate dispersion	Synpro Zincloid™	Ferro Chemical Co.
Zinc stearate	Synpro Zn stearate GP	Ferro Chemical Co.

Generic Name	Trade Mark	Vendor
Zinc stearate	Synpro Zn stearate wettable	Ferro Chemical Co.
Zinc stearate	Synpro Zn stearate S-1058	Ferro Chemical Co.

Aqueous Polyurethane Dispersion Preparation

For Example Nos. 1 and 13-23, a prepolymer was prepared by reacting 63.35 percent by weight (% w/w) VORANOL™ 5287 polyol, 33.3 %w/w ISONATE™ 50 MDI, 1.35  
5 %w/w diethylene glycol, and 2.00 %w/w polyethylene oxide monol having a molecular weight of 950. Example Nos. 2-6 used ISONATE™ 25 OP MDI for the precoat layer instead of ISONATE™ 50 MDI; the laminate layer was prepared with ISONATE™ 50 MDI. Example Nos. 13-18 were prepared with 1.63 parts per hundred resin of Paragum™ 241.  
Example Nos. 24 and 25 were prepared with a secondary backing. All layers were prepared  
10 with 5 parts per hundred of Nopco NDW.

The remaining steps were conducted at an ambient temperature of 19°C.

The prepolymer was fed continuously at a rate of 32.1 gallons/minute (121.5 liters/minute) through a first arm fitted to a first T. DeSulf DBS-60T surfactant was fed at a rate of 1.61 gallons/minute (6.09 liters/minute) through a first arm of a second T and merged  
15 with a water stream flowing at a rate of 5.5 gallons/minute (20.8 liters/minute) through the second arm of the second T. The prepolymer stream and the water/surfactant stream were merged at the first T and passed through a static mixer.

Next, they were fed to the input port of a IKA-SD 41 Super-Dispax™ dispersing

instrument (a trademark of Ika Works, Inc.). The IKA-SD 41 Super-Dispax™ dispersing instrument is a rotor/stator device, which was operated at 1200 rpm. The ratio of feeds into the dispersing instrument was 81.9% prepolymer, 4.1% surfactant solution, and 14.0% water.

A high internal phase ratio (HIPR) emulsion formed in the dispersing instrument. It  
5 had a volume average particle size of 0.285 microns and a polydispersity of 3.1 as measured by a Coulter LS130 particle size analyzer.

The HIPR emulsion from the dispersing instrument was fed into a first arm attached to a third T and merged with an aqueous stream fed through a second arm of the third T at the rate of 5.1 gallons/minute (19.3 liters/minute). The output of the combined streams was fed  
10 into one arm of a fourth T that was attached to the input of a Lightnin™ model .025 LB in-line blender (a trademark of Greey/Lightnin).

Chain extension was accomplished in the in-line blender by concurrently pumping a 10% aqueous piperazine solution through the other arm of the fourth T. The piperazine solution was added at a constant rate of 18.0 gallons/minute (68.1 liters/minute) (0.75  
15 equivalents based on the isocyanate groups of the prepolymer). The two streams were mixed in the in-line blender operating at 1500 rpm.

The product was collected and allowed to stand overnight to allow the water to react with the react with the remaining isocyanate groups. The resulting stable poly(urethane/urea) dispersion was found to have a solids content of 56.0% by weight and a volume average  
20 particle size of 0.256 microns and a polydispersity of 3.5 as measured by a Coulter LS 230 particle size analyzer.

Example No. 7 – Styrene-Butadiene Latex Preparation

A styrene-butadiene latex test sample was substituted in the polyurethane dispersion formulations. The latex contained approximately fifty- percent solids.

British Spill Test Carpet Coating Sample Preparation

5       The British Spill Test Carpet Coating samples were prepared using the following method. First, a carpet sample was stretched onto a frame. Next, the carpet was held in place on a tandem roll coater. A permanent marker was used to mark off an 11"x14" (28cm x 35.6cm) area on the carpet.

10      The amount of precoat and laminate to achieve the desired coat weight was determined using the following calculation:

- a) Calculate area to cover ( $\text{yd}^2$ ) = length \* width = 11" \* 14" = 154  $\text{in}^2$  \* (1  $\text{yd}^2$  / 1296  $\text{in}^2$ ) = 0.119  $\text{yd}^2$  (0.099  $\text{m}^2$ );
- b) Target DRY   38 oz/ $\text{yd}^2$  (900.7g/ $\text{m}^2$ )   80% precoat – 30.4 oz/ $\text{yd}^2$  (720.5g/ $\text{m}^2$ )  
20% laminate = 7.6 oz/ $\text{yd}^2$  (180.1g/ $\text{m}^2$ );
- c) Target WET   30.4 oz/ $\text{yd}^2$  (730.5g/ $\text{m}^2$ ) / 0.762 solids = 39.9 oz/ $\text{yd}^2$  (945.6g/ $\text{m}^2$ )  
7.6 oz/ $\text{yd}^2$  (180.1g/ $\text{m}^2$ ) / 0.762 solids = 9.60 oz/ $\text{yd}^2$  (227.5g/ $\text{m}^2$ );
- d) For precoat, convert oz to grams (39.9oz/ $\text{yd}^2$ ) (1lb/16oz)(454g/1lb) = 1132 g/ $\text{yd}^2$  (945.6g/ $\text{m}^2$ );
- e) Use calculated coverage area to give grams needed (1132 g/ $\text{yd}^2$ ) \* 0.119 $\text{yd}^2$  = 134.7g;
- f) Add 20g to account for coverage on the jumbo roller; and
- g) Repeat steps d-f for the laminate coat and add only an additional 10 grams for

jumbo roller coverage.

The roller was placed such that its milled gap and side bars were at the top of the carpet. The precoat was poured and distributed evenly in front of the roller. Without applying any pressure, the ends of the roller were held and rolled to the permanent mark at 5 the end of the carpet. The jumbo roller with two side weights was rolled from the bottom up and down two times.

The laminate was weighed out. The roller without the milled gap was placed at the top of the carpet. The laminate was poured and distributed evenly in front of the roller. Without applying any pressure, the ends of the roller were held and dragged to the permanent 10 mark at the end of the carpet.

The carpet was then clipped in place at the edges of the frame and immediately placed into a 400°F (204°C) oven until the pyrometer read a temperature of 265°F (129°C) on the carpet. The carpet was then removed from the oven and allowed to cool to room temperature. After the carpet was cooled, it was removed from the frame and placed in a controlled 15 environment to equilibrate.

Example Nos. 24 and 25 - Carpets

with Secondary Backing Preparation

The secondary backing carpet Example Nos. 24 and 25 were prepared using the following method. First, a carpet sample was held down at its top and bottom edges to a 20 Plexiglas surface. Next, a permanent marker was used to mark off an 11"x15" (28cm x 38.1cm) area on the carpet.

The amount of precoat and laminate to achieve the desired coat weight was

determined using the following calculation:

- a) Calculate area to cover ( $\text{yd}^2$ ) = length \* width =  $11'' * 15'' = 165 \text{ in}^2 * (1 \text{ yd}^2 / 1296 \text{ in}^2) = 0.127 \text{ yd}^2 (0.106 \text{ m}^2)$ ;
- b) Target DRY 44 oz/yd<sup>2</sup> (1042.8 g/m<sup>2</sup>) 80% precoat – 35.2 oz/yd<sup>2</sup> (834.2 g/m<sup>2</sup>)  
5 20% laminate = 8.8 oz/yd<sup>2</sup> (208.6 g/m<sup>2</sup>);
- c) Target WET 35.2 oz/yd<sup>2</sup> (834.2 g/m<sup>2</sup>) / 0.762 solids = 46.2 oz/yd<sup>2</sup> (1094.9 g/m<sup>2</sup>)  
8.8 oz/yd<sup>2</sup> (208.6 g/m<sup>2</sup>) / 0.762 solids = 11.1 oz/yd<sup>2</sup> (263.1 g/m<sup>2</sup>);
- d) For precoat, convert oz to grams (46.2oz/yd<sup>2</sup> )(1lb/16oz)(454g/1lb) = 1311 g/yd<sup>2</sup> (1096 g/m<sup>2</sup>);
- e) Use calculated coverage area to give grams needed (1311 g/yd<sup>2</sup>) \* 0.127y<sup>2</sup> = 166.5g;
- f) Add 20g to account for coverage on the jumbo roller; and
- g) Repeat steps d-f for the laminate coat and add only an additional 10 grams for jumbo roller coverage.

15 The roller was placed such that its milled gap and side bars were at the top of the carpet. The precoat was weighed out. The precoat was poured and distributed evenly in front of the roller. Without applying any pressure, the ends of the roller were held and rolled to the permanent mark at the end of the carpet. The jumbo roller with two side weights was rolled from the bottom up and down two times.

20 The laminate was weighed out. The roller without the milled gap was placed at the top of the carpet. The laminate was poured and distributed evenly in front of the roller. Without applying any pressure, the ends of the roller were held and dragged to the permanent

mark at the end of the carpet.

A drawdown box with its 25 mm side down was placed on a second piece of Plexiglas. The drawdown box was filled to  $\frac{3}{4}$  full with evenly distributed precoat. With a downward pressure, the drawdown box was used to put a film of the compound onto the  
5 second piece of Plexiglas.

A piece of secondary backing was cut to the size of the carpet and placed on top of the film. A roller was rolled over the backing to the end of the glass. The secondary backing was removed from the glass and placed over the coated area of the carpet such that coated area of the backing faced the coated area of the carpet.

10 The carpet and the secondary backing were held together with a weight at their top edge. The coatings were married together with a roller.

The carpet and the secondary backing were placed into a 275°F (135°C) oven until the pyrometer read a temperature of 265°F (129°C) on the carpet. The carpet was then removed from the oven and placed in a controlled environment to equilibrate.

15 Production Scale Example Nos. 8-12

A 3300-pound (1497 kg) batch of the precoat compound and a 3200-pound (1451 kg) batch of the laminate compound were prepared using standard compound mixing equipment. The finishing range consisted of a dual director applicator and a scrim pan-roll coater. The direct coaters were set up to split the precoat and laminate coatings to a desired 80-20  
20 distribution.

A first roll coater had a six inch (15.2 cm) diameter and ran at 30+ rpm. The second roll coater had an eight inch (20.3 cm) diameter and was maintained stationary. The adhesive

pan was as small as practical for the roll coater and auxiliary edge coater rolls.

The tufted carpet fibers through the primary backing was coated face down with the precoat roller speed set at the speed of the carpet and weight set at normal production rates. The laminate roller was fixed and gapped. A lick roll coater that positioned directly in the 5 adhesive pan was used to apply the precoat. The wet coating wet distribution was 40 opsy (ounces per square yard) ( $948 \text{ g/m}^2$ ) precoat, 10 opsy ( $237 \text{ g/m}^2$ ) laminate coat, and 8 opsy ( $190 \text{ g/m}^2$ ) adhesive coat. After marrying, the carpet with its face up was dried in a 400°F (204°C) oven.

Tables 1-4

10 Table 1 contains the formulation properties for Example Nos. 1-33. Table 1 also identifies the additives and inorganic fillers, if any, and the amounts added in parts per hundred resin.

Table 2 contains the physical properties of Example Nos. 1-7. Example Nos. 1-6 contained 200 pph filler loads in the laminate layers with non-treated calcium carbonate. 15 Example 4 also contained 25% stearic acid-treated calcium carbonate filler in the precoat. But, all failed the British Spill test.

Table 3 contains physical testing data for Example Nos. 8-12. Example Nos. 8-10 were non-treated calcium carbonate carpet samples, and Example Nos. 11-12 were prepared with stearic acid-treated calcium carbonate. The laminate layers contained 200 pph filler 20 loads. All of the non-treated carpet samples failed the British Spill test while both of the acid-treated samples passed.

Table 4 contains physical testing data for Example Nos. 13-33. Example No. 13 was

prepared with ammonium stearate. Example Nos. 14-18 were prepared with calcium stearate; Example Nos. 19, 24, 26, and 32 were prepared with an ammonium stearate/calcium carbonate combination; and Example Nos. 20-23, 25, 27-31, and 33 were prepared with a zinc stearate/calcium carbonate combination. Example No. 13 passed the British Spill test.

- 5 While Example No. 14 had no bleed through, it had a watermark. Example Nos. 15-18 passed the British Spill test. Example Nos. 14-18 demonstrate that calcium stearate generally provides good spill resistance as measured by the British Spill test.

Example Nos. 19, 24, 26, and 32 passed the British Spill test, but each had a watermark. Example Nos. 19, 24, 26, and 32 demonstrate that ammonium stearate provides  
10 good spill resistance as measured by the British Spill test. Similarly, Example Nos. 20-23, 25, 27-31, and 33 passed the British Spill test, but Example Nos. 20-23, 27-28, and 30-31 had a watermark. Significantly, Example Nos. 30 and 31 only demonstrated a small watermark after 5 hrs and Example No. 33 did not show a watermark. Example Nos. 20-23, 25, 27-31, and 33 illustrate that the combination of zinc stearate and calcium carbonate provides spill  
15 resistance as measured by the British Spill test.

Table 1

Example #	1	2	3	4	5	6
Precoat - Properties						
Filler Load	150	150	150	150	150	150
AFV <sup>1</sup> , #3@20rpm, cps	N/A	N/A	N/A	N/A	N/A	N/A
AF cup wt, g/3oz. (g/85g)	N/A	N/A	N/A	N/A	N/A	N/A
Compound Visc. (#5@20rpm), cps	16500	N/A	N/A	N/A	N/A	N/A
Compound Cup wt, g/3oz. (g/85g)	123.6	N/A	N/A	N/A	N/A	N/A
Additives	ATH <sup>2</sup>	None	ATH	25% CS11	MP <sup>3</sup>	5% DC85
Coat Visc. (#5@20rpm), cps	16340	10460	12300	12020	N/A	16100
Coating Cup wt, g/3oz. (g/85g)	126.1	132.3	118.6	108.6	N/A	129
Coat weight, oz/yd <sup>2</sup> (g/m <sup>2</sup> )		41.7 (988)	39.2 (929)	38.1 (903)	N/A	39.9 (946)
Laminate Coat - Properties						
Filler Load	200	200	200	200	200	200
Filler	GM 8 <sup>4</sup>	GM 8				
Filler Description	CaCO <sub>3</sub>					
Compound Visc. (#5@20rpm), cps	18800	19300	19300	19300	19300	19300
Compound Cup wt, g/3oz (g/85g)	N/A	N/A	N/A	N/A	N/A	N/A
Additives	None	None	None	None	None	None
Coat Visc. (#5@20rpm), cps	21800	21800	N/A	N/A	N/A	N/A
Coating Cup wt, g/3oz. (g/85g)	131.4	131.4	N/A	N/A	N/A	N/A

<sup>1</sup>AFV = after filler viscosity; <sup>2</sup>ATH = aluminum trihydrate; <sup>3</sup> MP = Michem P4983-40R;

<sup>4</sup>GM 8 = Georgia Marble #8 Calcium carbonate

Example #	7
Precoat - Properties	
Filler Load	150
AFV, #3@20rpm, cps	N/A
AF cup wt, g/3oz. (g/85g)	N/A
Compound Visc. (#5@20rpm), cps	12980
Compound Cup wt, g/3oz. (g/85g)	N/A
Additives	None
Coat Visc. (#5@20rpm), cps	13420
Coating Cup wt, g/3oz. (g/85g)	126.3
Coat weight, oz/yd <sup>2</sup> (g/m <sup>2</sup> )	38.2 (905)
Laminate Coat - Properties	
Filler Load	200
Filler	GM 8
Filler Description	CaCO <sub>3</sub>
Compound Visc. (#5@20rpm), cps	31450
Compound Cup wt, g/3oz (g/85g)	N/A
Additives	None
Coat Visc. (#5@20rpm), cps	29500
Coating Cup wt, g/3oz. (g/85g)	131.4

Example #	8	9	10	11	12
Precoat - Properties					
Filler Load	150	150	200	200	200
Additives					
Coat Visc. (#5@20rpm), cps	10360	10360	22350	28000	28000
Coating Cup wt, g/3oz. (g/85g)	124.5	124.5	140.2	143.1	143.1
Laminate Coat - Properties					
Filler Load	200	200	200	200	200
Filler	GM 8	GM 8	GM 8	CS11	CS11
Filler Description	CaCO <sub>3</sub>	CaCO <sub>3</sub>	CaCO <sub>3</sub>	Acid CaCO <sub>3</sub>	Acid CaCO <sub>3</sub>
Additives					
Coat Visc. (#5@20rpm), cps	18050	18050	22350	28000	28000
Coating Cup wt, g/3oz. (g/85g)	137.9	137.9	140.2	143.1	143.1

Example #	13	14	15	16	17	18
Laminate Coat - Properties						
Filler Load (Total)	200	200	200	200	200	200
Filler	None	Ca Stearate				
Filler Load (Ca Stearate)	0	1.0	1.0	2.0	5.0	10.0
Additives	STANFAX™ NH <sub>4</sub> Stearate .75phr <sup>5</sup>					
Coat Visc. (#5@20rpm), cps	23350	20200	22150	22700	24250	25700
Coating Cup wt, g/3oz. (g/85g)	125.79	129.60	132.00	139.50	134.50	126.70

<sup>5</sup>phr = parts per hundred resin

Example #	19	20	21	22	23	24
Laminate Coat - Properties						
Filler Load (Total)	200	200	200	200	200	200
Filler	NH <sub>4</sub> Stearate / GM 8	ZsD <sup>9</sup> / GM 8	ZsG <sup>10</sup> / GM 8	ZsW <sup>11</sup> / GM 8	ZsS <sup>12</sup> / GM 8	NH <sub>4</sub> Stearate / GM 8
Filler Load (Stearate Compound - NH <sub>4</sub> /Zn)	0.75/0	0/1.0	0/5.0	0/5.0	0/5.0	0.75/0
Additives						
Coat Visc. (#5@20rpm), cps	21350	16780	20100	20300	20250	21350
Coating Cup wt, g/3oz. (g/85g)	134.39	129.66	139.26	134.16	129.30	134.39
Coat weight, g/3oz (g/85g)	42.60	40.71	43.04	45.06	42.53	50.45

<sup>9</sup>ZsD = Zinc stearate dispersion; <sup>10</sup>ZsG = Zinc stearate GP; <sup>11</sup>ZsW = Zinc stearate wettable;

<sup>12</sup>Zinc stearate S-1058

Example #	25	26	27	28	29	30
Laminate Coat - Properties						
Filler Load (Total)	200	200	200	200	200	200
Filler	ZsW / GM 8	NH <sub>4</sub> Stearate / GM 8	ZsW / GM 8			
Filler Load (Stearate Compound - NH <sub>4</sub> /Zn)	0/5	0.75/0	0/1.0	0/3.5	0/5.0	0/10.0
Additives						
Coat Visc. (#5@20rpm), cps	20300	20450	2240	23200	20650	21050
Coating Cup wt, g/3oz. (g/85g)	134.16	129	139.4	135.5	133.1	132.1
Coat weight, g/3oz (g/85g)	50.71	40.08	43.15	41.94	41.40	40.87

Example #	31	32	33
Laminate Coat - Properties			
Filler Load (Total)	200	200	195
Filler	ZsW / GM 8	NH <sub>4</sub> Stearate / GM 8	ZsG / GM 8
Filler Load (Stearate Compound - NH <sub>4</sub> /Zn)	0/20.0	0.75/0	0/5.0
Additives			
Coat Visc. (#5@20rpm), cps	25900	24550	20550
Coating Cup wt, g/3oz. (g/85g)	130.3	137.37	129.9
Coat weight, g/3oz (g/85g)	40.98	44.81	42.12

Table 2

Ex.#	Tuft Bind (lb/kg)	Tuft Bind (Wet 20 min) (lb/kg)	%R.	Tuft Bind (Wet 4hr) (lb/kg)	Delam. (lb/in) (kg/cm)	Delam. (Wet 1Min) (lb/in) (kg/cm)	%R.	Delam. (Wet 4hr) (lb/in) (kg/cm)	Edge Ravel (Wet 4hr) (lb/kg)	Edge Ravel (204°C)	British Spill
1											
2	20 (9.08)				11.1 (1.98)	6.9 (1.23)		62.2			Fail @ 400°F (204°C)
3	22 (9.99)				11.2 (1.99)	7.2 (1.28)		64.3			Fail @ 400°F (204°C)
4	19.6 (8.90)				11.9 (2.12)	9.7 (1.73)		81.5			Fail @ 400°F (204°C)
5											Slight Fail @ 400°F (204°C)
6	20.2 (9.17)										Fail @ 400°F (204°C)
7	20.4 (9.26)										np @ 400°F (204°C), Blistered

R. = Retention; Delam. = Delamination; PH = Pinhole; wm = Watermark; Swm = Slight Watermark  
 VSwm = Very Slight Watermark; SM = Smudge; No BT = No Bleed Through; np = Near Pass

Table 3

Ex.#	Tuft Bind (lb/kg)	Tuft Bind (Wet 20 min) (lb/kg)	%R.	Tuft Bind (Wet 4hr) (lb/kg)	Delam. (lb/in) / (kg/cm)	Delam. (Wet 1Min) / (lb/in) / (kg/cm)	%R.	Delam. (Wet 4hr) / (lb/in) / (kg/cm)	Edge Ravel (Wet 4hr) (lb/kg)	Edge Ravel (Wet 4hr) (lb/kg)	British Spill
8	22.8 (10.4)			9 (4.09)	9.7 (1.73)			4.1 (0.73)	4.7 (2.13)	1.4 (0.64)	Fail
9	20.8 (9.44)			9.1 (4.13)	9.7 (1.73)			4 (0.71)	4 (1.82)	1.2 (0.54)	Fail
10	21.3 (9.67)			8.7 (3.95)	9.1 (1.62)			4 (0.71)	3.8 (1.73)	0.9 (0.41)	Fail
11	23.1 (10.5)			10.4 (4.72)	14.1 (2.51)			7.8 (1.39)	4.8 (2.18)	1.7 (0.77)	Pass
12	18.2 (8.26)			9 (4.09)	11.9 (2.12)			6.3 (1.12)	4.5 (2.04)	1.4 (0.64)	Pass

R = Retention; Delam. = Delamination; PH = Pinhole; wn = Watermark; Swm = Slight Watermark  
VSwm = Very Slight Watermark; SM = Smudge; No BT = No Bleed Through; np = Near Pass

Table 4

Ex.#	Tuft Bind (lb/kg)	Tuft Bind (Wet 20 min) (lb/kg)	%R.	Tuft Bind (Wet 4hr) (lb/kg)	Delam. (lb/in) (kg/cm)	Delam. (Wet 1Min) (lb/in) (kg/cm)	%R.	Delam. (Wet 4hr) (lb/in) (kg/cm)	Edge Ravel (Wet 4hr) (lb/kg)	Edge Ravel (Wet 4hr) (lb/kg)	British Spill
13	15.9 (7.22)	6.7 (3.04)									
14	19.1 (8.67)	6.8 (3.09)									
15	18.7 (8.49)	7.7 (3.50)									No BT, wm
16	17.2 (7.81)	7.8 (3.54)									
17	18.8 (8.53)	8.6 (3.90)									
18	18.3 (8.31)	9.8 (4.45)									
19	17.5 (7.95)	6.6 (3.00)									
20	16.4 (7.44)	4.9 (2.22)									
											Pass, wm
											Pass, vm

R. = Retention; Delam. = Delamination; PH = Pinhole; wn = Watermark; Swn = Slight Watermark  
 VSwm = Very Slight Watermark; SM = Smudge; No BT = No Bleed Through; np = Near Pass

Ex.#	Tuft Bind (lb/kg)	Tuft Bind (Wet 20 min) (lb/kg)	%R.	Tuft Bind (Wet 4hr) (lb/kg)	Delam. (lb/in) (kg/cm)	Delam. (Wet 1Min) (lb/in) (kg/cm)	%R.	Delam. (Wet 4hr) (lb/in) (kg/cm)	Edge Ravel (Wet 4hr) (lb/kg)	Edge Ravel (Wet 4hr) (lb/kg)	British Spill
21	16.2 (7.35)	7.4 (3.36)	45.7								Pass, VSwm
22	16.0 (7.26)	7.9 (3.59)	49.3								Pass, VSwm
23	16.5 (7.49)	7.7 (3.50)	46.8								Pass, VSwm
24	18.4 (8.35)	9.3 (4.22)	50.5								Pass, VSwm
25	19.6 (8.90)	14.0 (6.36)	71.5								Pass
26	17.2 (7.81)	7.1 (3.22)	41.2								Pass
27	20.5 (9.31)	8.1 (3.68)	39.6								Pass, wm
28	20.0 (9.08)	7.4 (3.36)	36.9								Pass, wm
29	17.0 (7.72)	9.0 (4.09)	52.9								Pass

R. = Retention; Delam. = Delamination; PH = Pinhole; wn = Watermark; Swm = Slight Watermark  
 VSwm = Very Slight Watermark; SM = Smudge; No BT = No Bleed Through; np = Near Pass

Ex.#	Tuft Bind (lb/kg)	Tuft Bind (Wet 20 min) (lb/kg)	%R.	Tuft Bind (Wet 4hr) (lb/kg)	Delam. (lb/in) (kg/cm)	Delam. (Wet 1Min) (lb/in) (kg/cm)	%R.	Delam. (Wet 4hr) (lb/in) (kg/cm)	Edge Ravel (Wet 4hr) (lb/kg)	Edge Ravel (Wet 4hr) (lb/kg)	British Spill
30	18.7 (8.49)	8.0 (3.63)	42.9								Pass, Swm @5hrs
31	19.0 (8.63)	8.5 (3.86)	45.0								Pass, Swm @5hrs
32	18.0 (8.17)	7.9 (3.59)	44.2								Pass, wm
33	16.5 (7.49)	10.3 (4.68)	62.1								Pass

R. = Retention; Delam. = Delamination; PH = Pinhole; wm = Watermark; Swm = Slight Watermark  
 VSwm = Very Slight Watermark; SM = Smudge; No BT = No Bleed Through; np = Near Pass

The embodiments described herein are given to illustrate the scope and spirit of the present invention. The embodiments herein will make apparent to those skilled in the art other embodiments that may also be used. These other embodiments are within the scope of the present invention. Thus, the scope of the invention should be determined by the 5 appended claims and their legal equivalents, rather than by the embodiments given herein.

**What is claimed is:**

1. 1. An aqueous dispersed polymeric composition for preparing a spill resistant carpet  
2. backing comprising:
  3. a. an aqueous dispersed polymeric material;
  4. b. an inorganic filler; and
  5. c. a hydrophobic compound selected from the group consisting of a hydrophobic  
6. acid, a salt of a hydrophobic acid, and mixtures thereof.
1. 2. The aqueous dispersed polymeric composition of Claim 1, wherein the aqueous  
2. dispersed polymeric material is selected from the group consisting of a polyurethane  
3. dispersion, a styrene-butadiene latex, a butadiene-acrylonitrile latex, an ethylene-vinyl  
4. acetate latex, a styrene-butadiene-butyl acrylate latex, a chloroprene latex, a  
5. polyethylene copolymer latex, an ethylene-styrene latex, a styrene-butadiene-  
6. vinylidene chloride latex, a styrene-alkyl acrylate latex, a vinyl latex, an acrylic latex,  
7. and mixtures thereof.
1. 3. The aqueous dispersed polymeric composition of Claim 2, wherein the inorganic filler  
2. is selected from the group consisting of calcium carbonate, calcium sulfate, kaolin,  
3. lignite fly ash, silica, talc, feldspar, mica, glass spheres, wollastonite, aluminum  
4. trihydrate, aluminum oxide, fiber glass, and mixtures thereof.
1. 4. The aqueous dispersed polymeric composition of Claim 3, wherein the hydrophobic  
2. compound is selected from the group consisting of butyric acid, hexanoic acid,  
3. octanoic acid, decanoic acid, dodecanoic acid, lauric acid, myristic acid, palmitic acid,  
4. oleic acid, linoleic acid, stearic acid, linolenic acid, gum rosin, wood rosin, tall oil

5       rosin, abietic acid, oxidized polyethylene containing carboxylic acid groups, ethylene-  
6       acrylic acid copolymers, ethylene-methacrylic acid copolymers, polyolefins grafted  
7       with unsaturated carboxylic acids, polyolefins grafted with anhydrides, methacrylic  
8       acid, maleic acid, fumaric acid, acrylic acid, and salts thereof.

1       5. The aqueous dispersed polymeric composition of Claim 4, wherein the aqueous  
2       dispersed polymeric material is a polyurethane dispersion, the inorganic filler is  
3       calcium carbonate, and the hydrophobic salt is zinc stearate.

1       6. An aqueous dispersed polymeric composition for preparing a spill resistant carpet  
2       backing comprising:

3           a. an aqueous dispersed polymeric material; and  
4           b. a treated inorganic filler, having been treated with a hydrophobic compound  
5       selected from the group consisting of a hydrophobic acid, a salt of a  
6       hydrophobic acid, and mixtures thereof.

1       7. The aqueous dispersed polymeric composition of Claim 6, wherein the aqueous  
2       dispersed polymeric material is selected from the group consisting of a polyurethane  
3       dispersion, a styrene-butadiene latex, a butadiene-acrylonitrile latex, an ethylene-vinyl  
4       acetate latex, a styrene-butadiene-butyl acrylate latex, a chloroprene latex, a  
5       polyethylene copolymer latex, an ethylene-styrene latex, a styrene-butadiene-  
6       vinylidene chloride latex, a styrene-alkyl acrylate latex, a vinyl latex, an acrylic latex,  
7       and mixtures thereof.

1       8. The aqueous dispersed polymeric composition of Claim 7, wherein the inorganic filler  
2       is selected from the group consisting of calcium carbonate, calcium sulfate, kaolin,

- 3        lignite fly ash, silica, talc, feldspar, mica, glass spheres, wollastonite, aluminum  
4        trihydrate, aluminum oxide, fiber glass, and mixtures thereof.
- 1        9. The aqueous dispersed polymeric composition of Claim 8, wherein the hydrophobic  
2        compound is selected from the group consisting of butyric acid, hexanoic acid,  
3        octanoic acid, decanoic acid, dodecanoic acid, lauric acid, myristic acid, palmitic acid,  
4        oleic acid, linoleic acid, stearic acid, linolenic acid, gum rosin, wood rosin, tall oil  
5        rosin, abietic acid, oxidized polyethylene containing carboxylic acid groups, ethylene-  
6        acrylic acid copolymers, ethylene-methacrylic acid copolymers, polyolefins grafted  
7        with unsaturated carboxylic acids, polyolefins grafted with anhydrides, methacrylic  
8        acid, maleic acid, fumaric acid, acrylic acid, and salts thereof.
- 1        10. The aqueous dispersed polymeric composition of Claim 9, wherein the aqueous  
2        dispersed polymeric material is a polyurethane dispersion, the treated inorganic filler  
3        is stearic acid-treated calcium carbonate.
- 1        11. A kit for preparing a spill resistant carpet backing comprising:  
2            a. an aqueous dispersed polymeric material;  
3            b. an inorganic filler; and  
4            c. a hydrophobic compound selected from the group consisting of a hydrophobic  
5        acid, a salt of a hydrophobic acid, and mixtures thereof.
- 1        12. The kit of Claim 11, wherein the aqueous dispersed polymeric material is selected  
2        from the group consisting of a polyurethane dispersion, a styrene-butadiene latex, a  
3        butadiene-acrylonitrile latex, an ethylene-vinyl acetate latex, a styrene-butadiene-  
4        butyl acrylate latex, a chloroprene latex, a polyethylene copolymer latex, an ethylene-

- 5        styrene latex, a styrene-butadiene-vinylidene chloride latex, a styrene-alkyl acrylate  
6        latex, a vinyl latex, an acrylic latex, and mixtures thereof.
- 1     13. The kit of Claim 12, wherein the inorganic filler is selected from the group consisting  
2        of calcium carbonate, calcium sulfate, kaolin, lignite fly ash, silica, talc, feldspar,  
3        mica, glass spheres, wollastonite, aluminum trihydrate, aluminum oxide, fiber glass,  
4        and mixtures thereof.
- 1     14. The kit of Claim 13, wherein the hydrophobic compound is selected from the group  
2        consisting of butyric acid, hexanoic acid, octanoic acid, decanoic acid, dodecanoic  
3        acid, lauric acid, myristic acid, palmitic acid, oleic acid, linoleic acid, stearic acid,  
4        linolenic acid, gum rosin, wood rosin, tall oil rosin, abietic acid, oxidized  
5        polyethylene containing carboxylic acid groups, ethylene-acrylic acid copolymers,  
6        ethylene-methacrylic acid copolymers, polyolefins grafted with unsaturated  
7        carboxylic acids, polyolefins grafted with anhydrides, methacrylic acid, maleic acid,  
8        fumaric acid, acrylic acid, and salts thereof.
- 1     15. A kit for preparing a spill resistant carpet backing comprising:  
2        a. an aqueous dispersed polymeric material; and  
3        b. a treated inorganic filler, having been treated with a hydrophobic compound  
4        selected from the group consisting of a hydrophobic acid, a salt of a  
5        hydrophobic acid, and mixtures thereof.
- 1     16. The kit of Claim 15, wherein the aqueous dispersed polymeric material is selected  
2        from the group consisting of a polyurethane dispersion, a styrene-butadiene latex, a  
3        butadiene-acrylonitrile latex, an ethylene-vinyl acetate latex, a styrene-butadiene-

- 4       butyl acrylate latex, a chloroprene latex, a polyethylene copolymer latex, an ethylene-  
5       styrene latex, a styrene-butadiene-vinylidene chloride latex, a styrene-alkyl acrylate  
6       latex, a vinyl latex, an acrylic latex, and mixtures thereof.
- 1     17. The kit of Claim 16, wherein the inorganic filler is selected from the group consisting  
2       of calcium carbonate, calcium sulfate, kaolin, lignite fly ash, silica, talc, feldspar,  
3       mica, glass spheres, wollastonite, aluminum trihydrate, aluminum oxide, fiber glass,  
4       and mixtures thereof.
- 1     18. The kit of Claim 17, wherein the hydrophobic compound is selected from the group  
2       consisting of butyric acid, hexanoic acid, octanoic acid, decanoic acid, dodecanoic  
3       acid, lauric acid, myristic acid, palmitic acid, oleic acid, linoleic acid, stearic acid,  
4       linolenic acid, gum rosin, wood rosin, tall oil rosin, abietic acid, oxidized  
5       polyethylene containing carboxylic acid groups, ethylene-acrylic acid copolymers,  
6       ethylene-methacrylic acid copolymers, polyolefins grafted with unsaturated  
7       carboxylic acids, polyolefins grafted with anhydrides, methacrylic acid, maleic acid,  
8       fumaric acid, acrylic acid, and salts thereof.
- 1     19. A method for preparing a spill resistant carpet backing comprising:  
2       a. mixing an inorganic filler with an aqueous dispersed polymeric material; and  
3       b. admixing a hydrophobic compound selected from the group consisting of a  
4       hydrophobic acid, a salt of a hydrophobic acid, and mixtures thereof.
- 1     20. The method of Claim 19, wherein the aqueous dispersed polymeric material is  
2       selected from the group consisting of a polyurethane dispersion; a styrene-butadiene  
3       latex, a butadiene-acrylonitrile latex, an ethylene-vinyl acetate latex, a styrene-

- 4           butadiene-butyl acrylate latex, a chloroprene latex, a polyethylene copolymer latex, an  
5           ethylene-styrene latex, a styrene-butadiene-vinylidene chloride latex, a styrene-alkyl  
6           acrylate latex, a vinyl latex, an acrylic latex, and mixtures thereof.
- 1   21. The method of Claim 20, wherein the inorganic filler is selected from the group  
2           consisting of calcium carbonate, calcium sulfate, kaolin, lignite fly ash, silica, talc,  
3           feldspar, mica, glass spheres, wollastonite, aluminum trihydrate, aluminum oxide,  
4           fiber glass, and mixtures thereof.
- 1   22. The method of Claim 21, wherein the hydrophobic compound is selected from the  
2           group consisting of butyric acid, hexanoic acid, octanoic acid, decanoic acid,  
3           dodecanoic acid, lauric acid, myristic acid, palmitic acid, oleic acid, linoleic acid,  
4           stearic acid, linolenic acid, gum rosin, wood rosin, tall oil rosin, abietic acid, oxidized  
5           polyethylene containing carboxylic acid groups, ethylene-acrylic acid copolymers,  
6           ethylene-methacrylic acid copolymers, polyolefins grafted with unsaturated  
7           carboxylic acids, polyolefins grafted with anhydrides, methacrylic acid, maleic acid,  
8           fumaric acid, acrylic acid, and salts thereof.
- 1   23. A spill resistant carpet backing prepared in accordance with Claim 22.
- 1   24. The spill resistant carpet backing of Claim 23, wherein the spill resistant carpet  
2           backing is a carpet layer selected from the group consisting of a precoat, a laminate  
3           layer, and a foam layer.
- 1   25. A method for preparing a spill resistant carpet backing comprising:  
2       a. treating an inorganic filler with a hydrophobic compound selected from the  
3           group consisting of a hydrophobic acid, a salt of a hydrophobic acid, and

- 4                       mixtures thereof; and
- 5       b.       mixing the treated inorganic filler with an aqueous dispersed polymeric  
6                       material.
- 1     26.   The method of Claim 25, wherein the aqueous dispersed polymeric material is  
2                       selected from the group consisting of a polyurethane dispersion, a styrene-butadiene  
3                       latex, a butadiene-acrylonitrile latex, an ethylene-vinyl acetate latex, a styrene-  
4                       butadiene-butyl acrylate latex, a chloroprene latex, a polyethylene copolymer latex, an  
5                       ethylene-styrene latex, a styrene-butadiene-vinylidene chloride latex, a styrene-alkyl  
6                       acrylate latex, a vinyl latex, an acrylic latex, and mixtures thereof.
- 1     27.   The method of Claim 26, wherein the inorganic filler is selected from the group  
2                       consisting of calcium carbonate, calcium sulfate, kaolin, lignite fly ash, silica, talc,  
3                       feldspar, mica, glass spheres, wollastonite, aluminum trihydrate, aluminum oxide,  
4                       fiber glass, and mixtures thereof.
- 1     28.   The method of Claim 27, wherein the hydrophobic compound is selected from the  
2                       group consisting of butyric acid, hexanoic acid, octanoic acid, decanoic acid,  
3                       dodecanoic acid, lauric acid, myristic acid, palmitic acid, oleic acid, linoleic acid,  
4                       stearic acid, linolenic acid, gum rosin, wood rosin, tall oil rosin, abietic acid, oxidized  
5                       polyethylene containing carboxylic acid groups, ethylene-acrylic acid copolymers,  
6                       ethylene-methacrylic acid copolymers, polyolefins grafted with unsaturated  
7                       carboxylic acids, polyolefins grafted with anhydrides, methacrylic acid, maleic acid,  
8                       fumaric acid, acrylic acid, and salts thereof.
- 1     29.   A spill resistant carpet backing prepared in accordance with Claim 28.

- 1    30. The spill resistant carpet backing of Claim 29, wherein the spill resistant carpet
- 2        backing is a carpet layer selected from the group consisting of a precoat, a laminate
- 3        layer, and a foam layer.

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 99/18672

**A. CLASSIFICATION OF SUBJECT MATTER**  
 IPC 7 D06N7/00 C08K13/02

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 D06N C08K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DATABASE WPI Section Ch, Week 198510 Derwent Publications Ltd., London, GB; Class A81, AN 1985-059878 XP002127669 & JP 60 017174 A (JAPAN SYNTHETIC RUBBER CO LTD), 29 January 1985 (1985-01-29) abstract	1-3, 11-13, 19-21
Y		25-27
X	DE 32 15 890 A (BASSERMANN & CO) 3 November 1983 (1983-11-03)	6-9, 15-18
Y	page 11, line 19 - line 26; claims; examples	25-27
	---	-/-

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

\* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
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Date of the actual completion of the international search

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# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 99/18672

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	<p>DATABASE WPI            Section Ch, Week 199628            Derwent Publications Ltd., London, GB;            Class A12, AN 1996-275882            XP002127670            &amp; RU 2 047 626 C (RESOURCES ECONOMY            PROBLEMS INST),            10 November 1995 (1995-11-10)            abstract</p> <p>---</p>	1,2,11, 12
A	<p>DE 19 57 159 A (FEIT, LEO)            27 May 1971 (1971-05-27)</p>	4,5,14
A	<p>page 1, line 1 -page 2, line 5; claim 1;            example 19</p> <p>---</p>	3,4,13, 14
A	<p>DATABASE WPI            Section Ch, Week 198722            Derwent Publications Ltd., London, GB;            Class A18, AN 1987-153590            XP002127671            &amp; JP 62 090376 A (JAPAN SYNTHETIC RUBBER            CO LTD), 24 April 1987 (1987-04-24)            abstract</p> <p>---</p>	1,11,19
A	<p>WO 97 00995 A (MINNESOTA MINING &amp; MFG)            9 January 1997 (1997-01-09)            page 2, line 1 - line 19            page 9, line 3 -page 10, line 15</p> <p>---</p>	6,15,25
A	<p>US 4 643 930 A (UCCI POMPELIO A)            17 February 1987 (1987-02-17)            cited in the application            column 3, paragraph 1; claims</p> <p>---</p>	1,11
A	<p>US 4 619 853 A (BLYTH RANDOLPH C ET AL)            28 October 1986 (1986-10-28)            cited in the application            column 2, line 55 - line 66; claims</p> <p>---</p>	1,11

## INTERNATIONAL SEARCH REPORT

Information on patent family members

Internat'l Application No  
PCT/US 99/18672

Patent document cited in search report		Publication date	Patent family member(s)		Publication date
JP 60017174	A	29-01-1985	NONE		
DE 3215890	A	03-11-1983	NONE		
RU 2047626	C	10-11-1995	NONE		
DE 1957159	A	27-05-1971	NL	6917439 A	24-05-1971
JP 62090376	A	24-04-1987	JP	1830847 C	15-03-1994
WO 9700995	A	09-01-1997	US	5888290 A	30-03-1999
			AU	704867 B	06-05-1999
			AU	5964596 A	22-01-1997
			CA	2225526 A	09-01-1997
			EP	0837963 A	29-04-1998
			JP	11509585 T	24-08-1999
US 4643930	A	17-02-1987	NONE		
US 4619853	A	28-10-1986	NONE		